Oil & Fat Industries

The Technical Journal of the Soap and Edible Oil Trades

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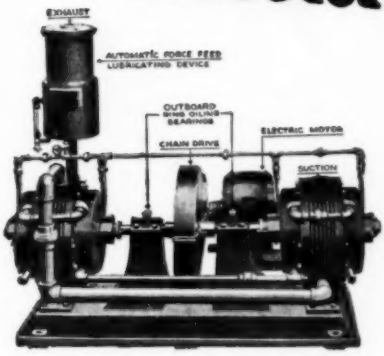

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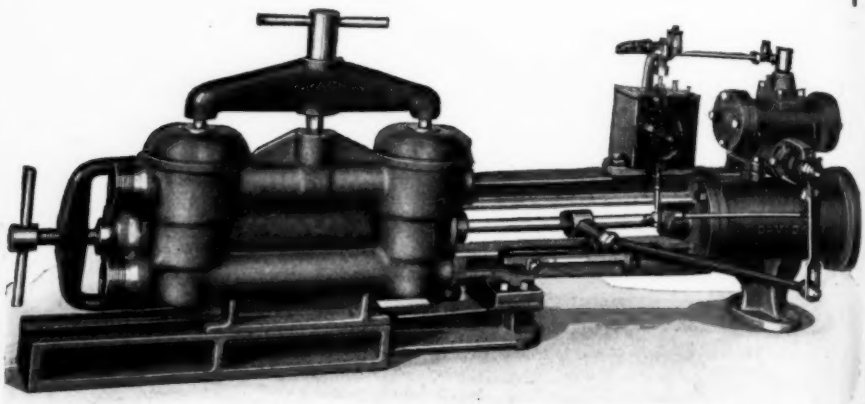

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OIL & FAT INDUSTRIES

VOLUME V SEPTEMBER, 1928 NUMBER 9

European Methods of Crushing Soya Beans

BY GEOFFREY E. GOVIER

PRIOR to 1908 Soya Beans were practically unknown to the European vegetable oil industry, but soon after the first experimental shipments of beans had been received from the Orient their great value as a source of excellent oil and meal was realized and it was not long before the prominent oil mills in most European countries were importing immense quantities.

The war gave the Soya Bean oil industry great momentum and record quantities were crushed between the years 1915-1920, both in Europe and the Orient, for during that period the latter was shipping very large quantities of crude oil to most of the European markets.

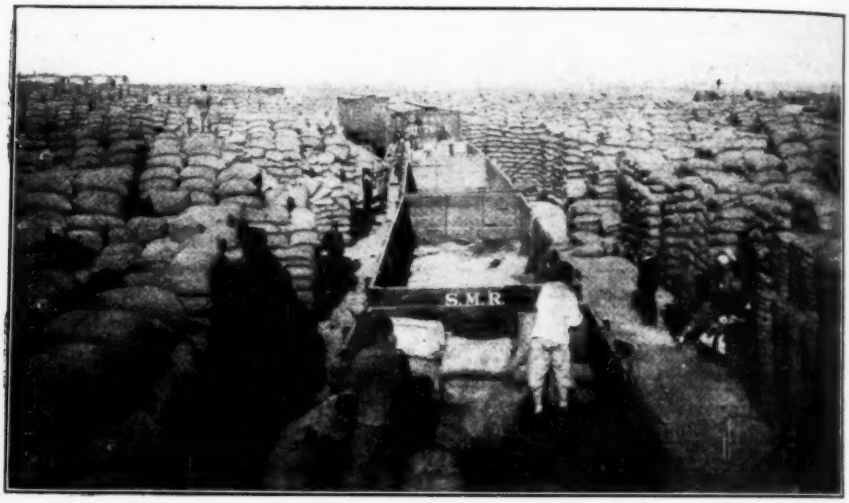
At that time the oil was not only used for technical purposes but a very considerable proportion of the output of the European mills was refined and hydrogenated and found its way into several edible products, such as margarine, lard substitute, suet substitute, etc.

Soya beans are still a very important factor in the European vegetable oil industry, and the oil is used very extensively by the soap industry; in the preparation of hard fat for candle manufacture; for "boiling" for use in paints as a substitute for linseed oil; also as a substitute for the latter in the manufacture of linoleum. During recent years a very extensive trade in edible soya bean oil has been developed in the South American countries, where the product is used both as a cooking and salad oil.

An interesting practice was adopted by one of the very prominent English oil mills during the war, when the idea was conceived of mixing the soya beans with cottonseed prior to processing. This produced an oil which was marketed as Soy-cot oil, and which, on refining produced a product of much lighter and more pleasing color than the straight soya bean oil, while it reacted very much more satisfactorily to hydrogenation. In addition to the improvement in the oil, the cake (also marketed as Soy-cot cake and meal) was much superior to neat cottonseed cake and became so popular, that Soy-cot products became one of the most important lines of this

particular mill (at that time rated as the largest plant of its kind in the world).

In the older European oil mills it was customary, when working soya beans, to grind the rolled beans under heavy edgestones before sending them to the kettles, but the modern practice is to entirely rely on rolling, which, when properly carried out gives as good results as grinding and eliminates the many objectionable features of edge stones. The thorough crushing and breaking of the oil cells is of vital importance in obtaining the best results.



Loading soya beans into cars at Dairen South Manchuria

Vertical-Pattern Rolls Popular

The writer found the vertical-pattern seed crushing rolls to be the most popular. Such rolls are made of chilled iron, which is preferred to the ordinary close-grained cast iron on account of its extreme hardness and consequent greater durability. These rollers are fitted with mild steel spindles, forced in with hydraulic pressure, the roller then being ground up true on its own spindle, and afterwards fluted if necessary. The cast iron housings have vertical slots in which the bearings of the upper rollers are free to slide. On the top of the housings is fixed a feed hopper of large capacity, fitted with a feed roller, by means of which a regular supply of beans can be fed into the crushing rollers. The quantity is regulated generally by a slide, but in some instances the writer saw special feeding devices being used.

The large rollers are driven at each end by fast and loose pulleys on the bottom roller, a more satisfactory system than driving rollers at one end only. It is said that those driven at each end run more steadily, wear more equally, and that the risk of fracture to the bottom roller driving shaft is reduced to a minimum.

A standard set of rollers has a capacity of two thousand pounds of soya beans per hour. A standard set is a set of rollers five feet high, each roller being 16 inches in diameter and 48 inches in

length. After having been passed through the crushing rolls the beans are sent to heating kettles. These kettles are used for heating and moistening the rolled beans, and the efficiency of this treatment is of great importance in getting the best results from the ultimate pressing. Both temperature and moisture require very careful adjustment. The temperature in some cases may have to be as high as 180°F, and to attain this, ample heating area and high steam pressure in the jacket are necessary, as it often occurs that steam cannot be added to the meal. For this reason the standard practice is to construct the kettles of steel, for there is less risk of failure and bursting than with cast iron; the conductivity of the plates being higher, a greater efficiency is obtained also. It is found to be better to use kettles of large capacity, for by treating as large a quantity of meal as possible in a batch a more uniform and thorough tempering of the meal is obtained. A double kettle is generally used when more than four presses are to be served from one kettle.

Cooking Methods

Moisture is added to the beans in the form of steam, which not only penetrates the meal thoroughly, but also helps to heat it. Special steam sprays are fitted to the kettles, designed to uniformly distribute the moisture. The are controlled by regulating valves with dials indicating the extent of opening, so that the operator knows just how much steam is being added to the batch. As the amount of natural moisture in the bean varies from time to time, it is important that control of the moistening apparatus should be simple, so that the ultimate moisture content may be kept constant, ensuring uniformity of the finished cake.

The tempered meal then goes to an automatic cake moulding machine. Four heating kettles serve one automatic cake moulding machine, and one of the latter serves sixteen Anglo-American hydraulic presses. One moulding machine averages two hundred and seventy cakes in thirty-five to forty minutes. A moulding machine is very easily operated. The operator places a cake tray and wrapper on the sliding carriage and then pushes this under the mould frame. The machine does the rest, and the operator has only to withdraw the carriage again with the moulded cake on it, and fold over the loose ends of the wrapper.

Description of Presses

The operator then places these cakes in the hydraulic press. In Europe, the Hydraulic Anglo Oil Press is now almost entirely used for expressing oil from Soya Beans. Generally a pressure of about four hundred tons is used, but where it is desired to make soya cake containing a minimum percentage of oil it is now usual to use heavier type presses with a gross pressure of from six hundred to eight hundred tons.

The number of cakes that can be made in a press is limited by the height a man can reach, and sixteen to eighteen has been found to be the best number for most purposes. Some exceed this number, but only by reducing the thickness of the cakes, or by making the height so great that there is difficulty in filling the upper plates, which results in loss of time and badly made cakes. Modern practice is to make the heaviest cake possible, so that on the newer

equipment ample space is allowed between the plates, so that thick cakes can be produced. This method enables the output to be increased without adding to the labor cost.

The size of the finished cakes is usually twelve to fourteen inches in width by thirty to thirty-two inches in length. The thickness and weight varies with the adjustment of the press, but it may be said that the average weight is fourteen pounds.

Arrange Press Capacity

The capacity of a sixteen cake press working on soya beans averages four hundred and fifty pounds per hour. A standard make of press has cylinders of cast steel, bored to standard gauges for rams, and, if carrying the columns, the holes for these are bored to jigs, as are the heads also, thus insuring accurate alignment of the columns. The rams are of close grained cast iron, ground smooth to avoid undue wear of leathers. Chilled rams are sometimes used.

The tables are of cast iron turned to fit the head of the ram and with corrugations on the upper face. The columns are constructed of mild steel, turned and polished with steel nuts top and bottom that bed over their whole area on the solid head and bottom, thus giving uniform stress on the columns. This gives a much stronger construction than columns with forged collars and loose caps on the head and bottom. The heads are of cast iron of box section, bored to jigs for reception of the columns, and with corrugations on the under side. In the lighter presses the bottoms are of cast iron, with large oil dishes to prevent the expressed oil from running into the foundation tanks below the presses, these tanks being sometimes entirely dispensed with. In the heavier presses the cylinder and bottom is one steel casting, the oil dish forming part of the casting, as in the lighter presses.

The plates are made of solid forged and rolled mild steel, with the usual corrugations formed during the rolling, thus giving strength and reducing breakages. Where special brands are required for the cakes, they are made of brass and inserted into suitable machined recesses in the plates.

Some manufacturers sell their whole output of cakes in the form that they leave the presses, others break them up into meal, for sale as straight soya bean meal, or make blends with other seed residues, molasses, dried meat, locust beans, etc., and produce special trade marked concentrates. The oil yield averages ten per cent. Very few soya beans are subjected to solvent extraction for it is found that the residual meal is unsuitable for feeding purposes.

American Linseed Oil Plants Sold

IN order to retire from the linseed oil business and to devote its entire attention to the food products trade, the American Linseed Company has sold its oil plants to the Archer-Daniels-Midland Company and Spencer Kellogg & Sons for a price not disclosed. The plants involved in the sale are the ones at Staten Island, Buffalo, Boston, Chicago, St. Paul, Portland, Ore., and Richmond. The capacity of these plants is 5,000,000 bushels of seed annually, and it is understood that each of the purchasing companies will get about one-half of the total properties passing under the sale.

Rumors of financial activity regarding the American Linseed Company have been prevalent for several weeks.

Ramifications of the Oil Industry

(Part I.) Processes*

BY THOMAS ANDREWS

Boiled and Settled Soaps

In boiled soaps the neutral oil or fatty acid is boiled and agitated with steam in the presence of caustic soda care being taken to see that the excess of caustic is not sufficient to cause the soap to grain, that is to say, that the solution of the soap in dilute caustic must not be impaired by making the solution too strong during the boiling. When the mass in the pan is in a state of chemical equilibrium it should consist of the soda salts of the fatty acids mixed with water, glycerol, excess of alkali, together with the unsaponifiable matter and the impurities in the oils.

The soap, not being soluble in salt solution, is thrown out of solution by the addition of salt to the pan. After settling for some time the soap will be found at the top of the pan as a curd and the bottom layer will consist of the added salt as well as the other matters referred to above. After the removal of this lower layer the soap is brought into solution again by the addition of enough water and is boiled with open steam. An excess of caustic is again added to complete the saponification and boiling is continued until there is no further absorption of caustic. The curd is again thrown out of solution and the bottom layer drawn off after a further settling period. This is repeated, with the addition of resin soaps if desired, and then the soap is closed with sufficient water to form a settled soap and yet be sufficiently open to allow of the discharge of the im-

Ewing Galloway

Frames in which soap is cooled and hardened

* Continued from August issue, *Oil and Fat Industries*.

purities. This fitted soap is allowed to remain at rest for several days during which time it settles out into three layers, namely; the settled soap at the top, the nigre or impure soaps at the middle and the foots at the bottom.

The top layer is the true settled soap and consists of about 63% fatty acids combined with about 7% of Na_2O, the remaining 30% being chiefly water.

The nigre is a layer of much diluted soap containing practically the whole of the insoluble soaps, the excess of caustic and the salt. Cooling forms are alkaline layer.

The settled soap is agitated with any medicants, colors or perfumes it is desired to incorporate in the soap until the whole is homogeneous and the mass is then cooled. Until recently the soap was cooled in box like frames, each frame holding about 10 cwt. of neat soap, and the cooling occupied many days. More recently it has become more usual to cool the soap in machines resembling the distance frame type filter press, where the hot soap may be considered as being in the distance frames and the plates are cored to allow of the passage of the cooling liquid, the soap being thus quickly cooled in thin layers. Whether the texture of thin quickly cooled soap can be equal to that obtained by the older method is a point open to discussion. If we are of the school that considers that the 30% of water in the settled soap is in the nature of water of crystallization we shall probably favor the old fashioned type of frame, whereas if we favor the view that the soap is in a colloidal solution we shall favor the quicker cooling machine. Each method has its adherents and such a discussion is outside of our scope herein.

Ewing Galloway

Stripped frames of soap

If fatty acids are used in the manufacture of the soap then it is possible to dispense with the dearer caustic soda and use the carbonate. Care must be taken,

however, to see that the solution and the fatty acids are brought into contact in such a manner that there is no loss through the evolution of the CO_2 from the carbonate. This evolution of gas is such a nuisance that the method is seldom used in spite of the cheapness of the carbonate. In the manufacture of soft soaps some potassium carbonate is intentionally added although there is very little to gain from a price consideration.

A good soft soap will contain all the glycerine in the original oil but there is, as a rule, only about 38% of fatty acids.

The addition of resin soaps to a settled soap cannot be looked upon as in the nature of adulteration as the alkaline salts of the resins have marked detergent properties and nearly the whole of the laundry soaps and soft soaps, not intended for use in the textile trades, contain some proportion of resin.

The dry soaps are chiefly powdered soap which has been dried and to which has been added some proportion of soda ash. Generally some inert scouring material is also added as well as one of the milder bleaching agents.

Glycerol Recovery

We have noted that after the first boiling together of the fatty matter and the caustic there is removed in the aqueous layer under the soap the bulk of the glycerine that was in the original oil together with the salt, the excess of caustic and the unsaponifiable matter that was in the oil. These liquors are known as soap lyes and are the chief source of our glycerine supply. These lyes may contain anything from 8%-10% of pure glycerol but before anything can be done with them it is necessary to remove the impurities.

The lyes are first treated in such a manner that the whole of the sodium salts (as far as is possible) is converted into the chloride, the fatty acids freed and the mucilage precipitated. This is carried out in practice by adding hydro-

Ewing Galloway

Kettle room in a large soap factory

chloric acid until the whole of the liquor is neutral or just faintly acid. At the same time, or as a later purification, chemical sand or one of the other albuminoid precipitating agents can be used to remove the mucilage. The liquor after being filtered can be further worked up for the removal of other impurities or may be evaporated to produce 80% soap lye crude glycerine. As the evaporation proceeds the saturation point of the brine solution is soon reached with the result that the salt is deposited within the evaporator. As it is usual to evaporate the solution under vacuum some method is necessary in order to remove the salt which is deposited, before it can seriously interfere with the heating surface of the evaporator. As the modern evaporator is designed to work continuously it is necessary to effect this without the loss of a large quantity of glycerine and without stopping the process. This is done by catching the salt as it falls out upon a grid within a box at the bottom of the evaporator so arranged that it can be isolated from the evaporator proper. When isolated the salt can be washed and the wash waters returned to the evaporator. The washed salt can then be dried with steam and dropped out of the box to be used again in salting out further batches of soap. The isolating valve is then re-opened so that further salt can drop into the box and the operation is repeated.

The further concentration of the glycerine is carried out by distillation in vacuo with the aid of superheated steam. The distilled product is condensed in a special arrangement of fractionating tubes and the fractions may consist of any dilution with water ranging from solid crystalline glycerol to pure water, the fractions depending to a large extent on the skill of the operative. The various fractions are concentrated to about 1.262 gravity and, provided the necessary quality is reached, constitute the dynamite glycerine of commerce.

Sulphonation Products

While on the subject of saponified oils I feel that some reference should be made to the "turkey red" oils. If castor oil is treated with fairly concentrated sulphuric acid at low temperatures in such a way that no sulphurous acid fumes are evolved the product after washing is a sulphoricinoleic acid and is found to consist, commercially, of two parts—a water soluble part and a water insoluble part—the sulphoricinoleic acid being the water soluble part. The two combined have the property of being miscible with water in all proportions and the solution will lather much in the same manner as a soap solution will lather. In this condition it is used for the cleansing of textile goods. The sodium and ammonium salts of the sulpho-acid are used as mordants in dyeing. These are made by adding solutions of sodium carbonate, caustic soda and ammonium hydrate to the sulphonated oil until the solution is just neutral or faintly acid. Those oils to be used in the mordanting of textile goods are generally neutralized with a mixture of soda and ammonia. These are generally sold as a solution in water and their value depends on the percentage of sulpho-ricinate present. The commercial preparations are generally viscid transparent fluids, their color depending on the quality of the oil from which they were prepared and also upon the temperature control that

has been exercised during the course of their preparation.

Fat Saponification

In the consideration of the oils for soap making it has been shown that the neutral oils are esters of glycerol and fatty acids. The splitting of the oil into these two parts constitutes a very large part of the oil industry.

There are four methods in use:

1. Splitting the neutral fat by steam pressure either in the presence of bases or not.
2. Splitting the neutral fat with the aid of sulphuric acid.
3. Splitting the neutral fat with the aid of sulpho-aromatic fatty acids.
4. Splitting the neutral fats by the aid of ferments.

In the first method steam at high pressure is blown through the oil or fat with which about 30% of water has been incorporated. During the process sufficient steam is allowed to escape from the autoclave, in which the reaction is carried out, to allow the mixture to be kept in a state of turmoil. If steam is used without the addition of a base a greater temperature will be required and consequently the fatty acids will be darker in color and the loss of glycerine will increase greatly. Moreover it is found that the amount of dissociation of the free fatty acid increases and the time taken to obtain a reasonable split is longer when splitting without a base. Works economy has decided that a split yielding about 98% of free fatty acids in the total fatty matter is the most efficient and this is very difficult to obtain when relying on plain aqueous saponification but occurs at about the eighth hour of steaming with the majority of fats when working with a steam pressure of 120 lbs. per sq.

Ewing Galloway

Modern automatic soap-press

in. with 3% of lime as the base. Other bases such as magnesia and zinc oxide have from time to time been used but lime has held its own in the majority of cases. This method is applicable to practically all oils and fats other than castor.

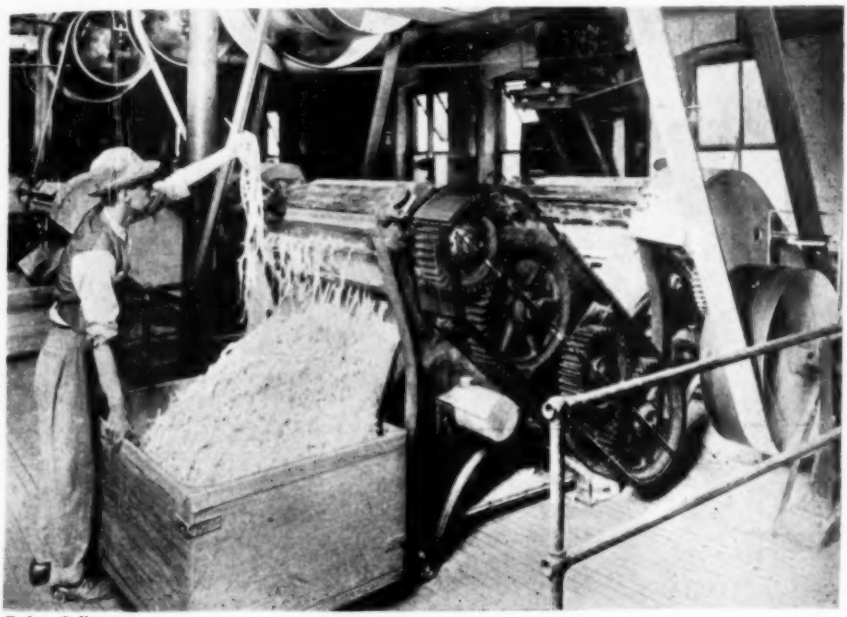
With regard to method number two. It will be remembered that in an earlier part of this paper reference was made to the formation of sulpho-acids by the action of strong sulphuric acid on neutral oils provided conditions favorable for the dissociation of the acids were absent. These sulpho-acids are readily hydrolyzed in the presence of boiling water giving the original fatty acids present in the oils plus some hydroxy fatty acids, some iso-oleic acid and the original glycerol. The sulphuric acid used in sulphonating is recoverable at the end of the reaction. This method of saponification is made use of to a large extent in the candle industry as owing to the formation of the solid iso-oleic acid and hydroxy-stearic acid the yield of candle material is increased. This increase as compared with aqueous saponification is considerable and may rise to as much as 12%.

Method number three is practically a sulphuric acid saponification but it is carried out in such a manner that the actual acid causing the saponification is in such dilute solution that the discoloration of the fatty acids is practically abolished. To revert again to the manufacture of our sulpho fatty acids, if, in the manufacture of them, we had added an aromatic compound such as benzene or naphthalene to the oil prior to the addition of the sulphuric acid we should have formed a mixed sulpho-compound of our fatty acid and the aromatic compound. We should, however, have found that the reaction would have been reversible in the presence of a low glycerine dilution. If on the contrary we had started with a *fatty acid* and an aromatic compound we could have formed the sulpho-aromatic compound in a more or less pure condition. In the presence of water and a trace of mineral acid these sulpho-compounds are capable of releasing nascent sulphuric acid, the reaction being reversible. Therefore if an oil is intimately mixed with one of these bodies in the presence of boiling water and some sulphuric acid we find that the nascent sulphuric acid is capable of saponifying the oil with the formation of the free fatty acid and glycerol. As there is present a comparatively large body of water the acid is liberated in a great excess of water and this affects the fatty acids in two ways. As the acid is dilute no dehydrating effect is obtainable and consequently the color of the oil does not suffer, but on the other hand for exactly the same reason, there is no increase in the "candle material" as no iso- or hydroxy-acids are formed.

With regard to method number four, it is common knowledge that seeds allowed to remain exposed to warmth and moisture give an oil higher in fatty acids than would have been obtained from the same seeds in a fresh condition. This led to the assumption that there was some hydrolyzing agent present in the seed. Research proved that this was the case, the enzyme was isolated and, commercially, it was found that, in particular, castor seed contained a ferment that was capable of splitting oils fairly successfully. The

ferment is obtained in various ways from the fresh seed and is capable, on being added to an emulsion of the oil and a dilute acid, of causing hydrolysis, yielding fatty acids and glycerine. The ferment is killed at temperatures of 40°C and over and cannot, therefore, be used in the production of fatty acids of high titre. Moreover the reaction is reversible at high splits making it necessary to remove the glycerine waters fairly frequently. There is a further objectionable feature in that, lying between the glycerine waters and the fatty acids, we find, on settling the whole, a middle layer consisting of an emulsion of fatty acids, glycerine, water and the ferment extract. It is possible to efficiently split castor oil with this process and I have obtained over 90% splits on castor oil, using "activators," on a works scale.

The glycerine that is obtained by any of these methods is ultimately worked up into dynamite glycerine in a similar manner to the lyes from the soap industry. The fatty acids when of a good color can be worked up into soap but the greater part are used in the manufacture of candles. No process gives a fatty acid that can be used in the candle industry direct owing to the poor color of all such acids, and to obtain the necessary light color and, at the same time, to remove the unsaponifiable matters and other impurities which would influence the burning of candles, the mixed fatty acids are distilled. The fatty acids are fed into a vacuum still and the temperature is raised, generally over an open fire, until the fatty acids are at the desired temperature. Superheated steam is then blown into the hot mass. The distillate con-



Ewing Galloway

Milling toilet soap

sisting of water and free fatty acids is fractionated in a series of tubes. At first the acids distill over freely and are of good color but as the proportion of foots increases in the still the color becomes worse and impurities increase. At this stage the contents of the still are taken out to be worked up with other quantities of foots. These distilled fatty acids consist of a mixture of liquid and solid acids. These are seeded at the right temperature so that crystals of the solid fatty acids lie in a mother liquor of the liquid fatty acids. In this state they are pressed to remove the liquid acids and the solid acids go forward to be used in the manufacture of candles.

Before we are in a position to carry out the various processes mentioned in the preceding part of this paper it is necessary to remove those impurities that will interfere with the process in any way. This calls for a multiplicity of refining processes and it is obvious that what is refining for one class of oil would be an absurdity in another. Just as an example I might quote the refining of an oil for edible purposes and the refining of the same oil for acid saponification. In the one case, that of edible refining, the refining would need to be carried out in such a way that the fatty acids were reduced to a minimum. Were this to be done to that portion of the oil that was to be saponified we should be removing from the oil that part which we require and at the same time we should find that the refining had considerably increased the time that would be taken in carrying out the process. The particular type of refining necessary and the impurities that would injure a given process form the bases of one of the largest departments in an oil works and beyond saying that the refinery often makes or mars the resulting products of other departments I am afraid that I can take this matter no further herein.

I have referred once or twice to the removal of the solid portion from the liquid portion of an oil. This is accomplished by cooling the oil until the solid portion "seeds" or crystallizes out of the liquid portion. The two are stirred together in those cases where the solid portion is a small fraction of the whole and are pumped through a filter press, the solid portion being retained as a cake. In the case of those oils and fatty acids where the solid portion is larger than the liquid portion the seeded material is placed in porous mats between the plates of hydraulic or screw presses and the liquid portion is squeezed out, the pressing being finished hot or cold according to the melting point desired.

Summary

In this paper I have endeavored to show the scope that is afforded by a study of the chief processes used and to give some idea of the many and varied aspects from which the oil industry may be viewed. Incidentally I have tried to show that varied as the processes may appear there are only a few fundamental facts on which the whole industry is based.

I would like to add that the modern oil works is run as a highly scientific co-ordinate whole and to say that it is no unusual business to sell ahead finished oil products, for which the oil seed even is not bought, under close guaranties of cost, quality, and delivery.

(*To be continued*)

Crude Cotton Oil Filtration

The Effect on Cottonseed Oil Refiners of Advanced Mill Practice

BY JOHN P. HARRIS AND BERNARD N. GLICK

FOR many years intelligent cotton oil refiners have wished that crushing mills would filter their crude oil and thereby remove from it all meal and suspended matter. They knew that this would largely do away with souring of the oil during shipment, as well as with tank bottoms in the tankcars received, both of which lead to claims, litigation, ill feeling and cleanup operations which have no proper place in vegetable oil production and refining.

Progressive refiners have long recognized that better crude oil, as pure as possible, means better shortening, cooking and salad oil, and therefore a wider distribution for their products in all fields, yet the industry, as a whole, offered no inducements to the crude miller to improve his oil until last season, and it so happened that the benefits of filtration were at an absolute minimum last season, owing to an unprecedented run of seed of excellent quality.

Advantages Conceded

We have yet to talk with any technically trained man connected with the industry in any way and familiar with its problems, who will deny the advantages of filtration to the crude miller and to the refiner.

Yet there is such a paucity of technically trained men connected with crude cotton oil milling and the habits of years are so difficult to overcome, that there is great apathy evidenced toward the idea of filtration. Many mill men apparently believe that the idea is useless, and strangely enough, many otherwise well informed refinery men are still under the impression that filtration is desirable but impractical.

To induce the crude oil miller to filter his oil, you must show him direct, tangible, day to day profits resulting therefrom. We personally are convinced, after handling 120 tank cars of oil made during the last crush, that straight filtration is desirable and indirectly profitable even if no purely adsorptive activated carbon were used. But, we have proven that the use of such an adsorptive carbon goes further than straight filtration, and yields these desired immediate profits with the certainty that is needed to induce the oil miller to filter, and we have set forth these facts in articles published in OIL & FAT INDUSTRIES and the *Cotton Oil Press*. We are directly responsible for those filtering installations which have been made in crude mills, and we take pride in them as a progressive contribution to this great industry.

Refiner Gains with Miller

There exists, however, in some quarters, a belief that the benefits of such filtration are limited to the crude mills at the expense of the refiner, and we wish to correct this impression by showing that the refiner actually gains just as much as does the crude oil miller.

This article, therefore, is direct-

ed to the refiner, and it is not our purpose to discuss the benefits of straight filtration of crude oil, because, as we understand it, this is not questioned by any refiner, and meets with his full approval, nor is it our purpose to explain the advantages of filtration to the crude mill, because such explanation has already been fully set forth in the August issue of the *Cotton Oil Press*, page 39. We shall simply explain here the advantages accruing to the refiner that result from the use of activated carbon in the filtration of crude oil.

Carbon Aids Filtration

A purely adsorptive, activated carbon has been extensively used for the last twenty years in the purification of syrups, oils, acids, glycerine, alkaloids, chemicals; in fact, there is hardly a branch of chemical industry where it is not regarded as a standard medium for purification. Its use involves filtration and, as a result, wherever filtration is one of the means necessary to the manufacture of any product, the addition of the carbon involves no increase in the cost of handling, helps filtration, and insures a far more complete purification than is possible with either straight filtration or filtration with a filter-aid, alone.

Let us concede that filtered crude oil is in every way desirable to the refiner. Experience has shown that straight filtration of crude is impossible owing to the slimy nature of the suspended matter to be removed, and it is necessary to use some sort of filter-aid, usually a variety of diatomaceous earth. Then since filtration is accepted why not use carbon to get further purification than is possible with straight filtration, involving as it does no extra expense outside of the negligible cost of the carbon.

Every refiner knows that in all crude cotton oil there exist certain colloids which cannot be removed by ordinary filtration, or filtration plus an inert filtering material. He also knows that during the refining operation these colloids are the direct cause of the emulsification of 5 percent to 15 percent of good, edible oil into the soap stock, according to the class of oil treated.

We want every refiner to know that the only action of activated carbon on crude oil is to adsorb and remove from the oil as much of these colloids as possible.

Refiners think of carbon as the most powerful of decolorizing agents because, in their refineries, at 160-220° F., it acts as such upon refined oil. But, if they were to try it at 90° F. to 100° F., (the temperatures used in crude oil filtration), they would find that its bleaching power was practically nil, and it is even less effective in this respect on crude oil.

Proof positive that this statement is true, is found in the fact that test after test has been run in both factory and laboratory upon refined oil produced from carbon filtered crude against refined oil from the same crude unfiltered, and the refined oil produced from carbon filtered crude invariably shows bleachability equal to or greater than that produced from unfiltered crude. For example, if a 7.6 red refined oil from unfiltered crude oil bleached down to 2.5 red, then a 7.6 red refined oil from carbon filtered crude will bleach down to 2.5 red, or better. We invite all refiners to make such tests in their own laboratories to prove to themselves that *carbon does not bleach crude oil!*

It is true that the refined oil from a carbon filtered crude does

have a distinctly lower refining loss than could be obtained from the same oil unfiltered, but this is due to exactly the same reasons that make good seed give a better oil than bad seed, i.e., less impurities in the oil.

It is also true that the refined oil from a carbon filtered crude will, *after refining*, give a lighter colored refined oil, but this is not due to the fact that the carbon has bleached the crude oil, but because it has removed from the crude oil some of those impurities which even the refining operation will not remove.

It is entirely wrong to refer to carbon filtration as a "partial" refining, or to insinuate that it interferes in any way with the legitimate province of the refiner. The carbon simply does for crude oil what it has done for other products during the last twenty years; it purifies the oil by extracting from it those extraneous and objectionable impurities that impair its value, both to the miller and the refiner.

This purification, moreover, results in the following desirable benefits to the refiner:

1. By elimination of moisture and impurities, it stabilizes the crude oil, so that it will not sour or decompose in transit. Thus, we may confidently expect perfect agreement in the sampling and analysis of the crude as loaded and at destination.

2. Such purified, sweet crude is certain to produce very much better finished products from the refinery, for, regardless of how good the refining equipment may be, it is certain that the best finished products are to be expected *only when the best grade of crude oil is used in the refinery!*

3. It is an unwritten law in the refinery, that crude cotton oil must be refined *immediately* upon receipt, the same day, if possible, and since deliveries generally pile up at the end of the month, due to a milling period which condenses the year's refining operations into a few months, it means that a refinery must be of large enough capacity to refine the entire season's crude in a short time. Even so, this large capacity often becomes inadequate at first of the month delivery periods, when it is often necessary to operate night and day in a feverish scramble to clear the track to avoid demurrage, and to refine the crude promptly. Such over-production periods are always costly from a labor standpoint and mean higher refining losses, through lack of proper settling time, spills, and lack of personal direction by the refiner in charge. Carbon filtered crude oil may be stored with safety and refined when convenient, with no danger of deterioration. Thus, the hectic, crowded periods of first of the month "refining sprees" become a thing of the past, and refinery operations may be spread over the entire year, increasing the capacity of every refinery.

4. Refiners all know that carbon has a distinct deodorizing effect on oils and that by using it on the crude, it will tend to remove from the oil as early as possible, a large part of the impurities that would give trouble in the deodorizers later.

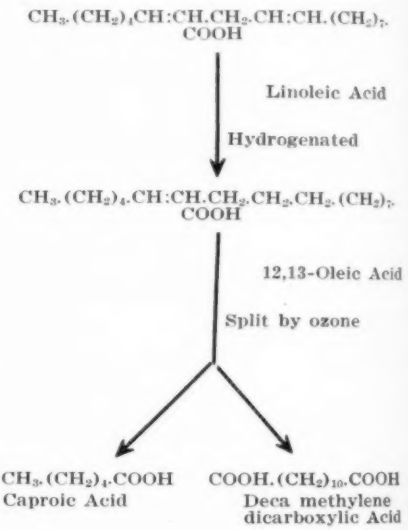
Summing up, we really feel that the general adoption of activated carbon in the filtration of crude oil will not only benefit the crude oil miller but will lead to large economies in refining.

Recent Work on Iso-Oleic Acid*

BY K. H. BAUR

THE designation "iso-oleic acids" is applied to all isomeric oleic acids which have the double bond of the eighteen carbon atom chain at some other place in the chain instead of between the ninth and tenth carbon atoms. Of the iso-oleic acids in natural oils, up to the present only the 6,7-oleic acid, petroselinic, has been found in parsley seed oil. The constitution of this acid has been elucidated by Vongerich and Eibner. The other members of this series have mostly been prepared synthetically. Iso-oleic acids have gained considerable interest as they occur and have been detected in hardened oils. They differ from oleic acid in that they are solid at ordinary temperatures, and are obtained as insoluble lead salts when the mixture of lead salts of both acids are extracted with alcohol or ether.

Some time ago it was attempted to separate the solid and liquid fatty acids, and isolate the iso-oleic acid by a combination of Twitchell's method with the brom-ester method of Grün and Jankow. This attempt was afterward abandoned. It was then attempted to break down the solid unsaturated acids of a hardened peanut oil to determine their composition. Baur and Mitsotakis separated the solid fatty acids in the form of their methyl esters by Twitchell's method and subjected them to splitting by means of ozone. On working up the split products deca methylene carbonic acid and caproic acid were obtained, so that the iso-oleic acid appears to exist in the hardened peanut oil as a 12,13-oleic acid. Such an acid can be obtained by hydrogenation of linoleic acid in such a manner that only the double bond in the 9,10-position will be hydrogenated, whereas that in the 12,13-position will remain unchanged. This is evident from the following structural formulae:



This discovery is an elaboration of the observation of Kaufmann who has proven by similar means that the hydrogenation of oils takes place first at the expense of linoleic acid.

*Reported and translated by the Universal Trade Press Syndicate. Read at the General Meeting of the German Chemical Society at Dresden, May 31, 1928.

Vitamins and Vital Elements

Suggesting a Possible Chemical Basis for Vitamin Activity

BY JOHN E. RUTZLER, JR.

THE vitamins are undetermined chemical substances which have been found to be indispensable to the normal functions of all mammalian, and of certain avian bodies. They are known by the effects which they produce. How the effects are produced is still a matter of conjecture.

The etymology of the word vitamin is quite obvious. Certain substances which could be isolated, but not in the pure state, have been found to be vital to the normal functions of the body. Hence, we have the word vital. Further, it was found that carbon, hydrogen, nitrogen, and oxygen were present in some of these substances. From this fact, and from the nomenclature of organic chemistry, the word amine came about. Combining these two, we have vital-amine. From this comes vitamine. But, since the insidious, habit forming alkaloids such as strychnine, morphine, and cocaine, nicotine, cinchonine, and brucine, end in ine, and since the vitamins do not possess any of the characteristics of these drugs, the final e of vitamine has been dropped.

Real Start in 1720

The history of the vitamins, as such, dates back to 1911, when Funk first introduced the name. However, the real beginning came in 1720, when Kremer wrote that medicine and surgery can be of no avail in cases of scurvy. He also suggested that scurvy can be cured by the use of green vegetables and certain fruit juices. Thus, by the inductive method, vent was given to the feeling that there was an important factor in nutrition, theretofore unrecognized.

Fruit Juices Treatment for Scurvy

What of the experimental? A few years later Lind contributed partial experimental verification of Kremer's tenet. Lind was the surgeon on the ship Salisbury. During a certain voyage, he had twelve scurvy patients. There was not sufficient orange and lemon juice for all the sufferers, so to ali of the men he gave the best care that circumstances permitted. To two of the patients he gave the juices of oranges and lemons, while to the others he gave different medicaments then advocated for the treatment of scurvy. Those who were given the fruit juices improved rapidly. The others were not affected by the care and feeding given them. Thus, the truth of Kremer's writings was substantiated in some measure.

Budd, in 1847, made further inductions along the same line, but to those pioneers the whole thing was entirely undecipherable.

The British navy introduced a regular ration of lemon juice in 1804. After that scurvy was very rare among Britain's naval force, while a few years prior thousands of cases were reported annually.

In 1885 the Japanese navy took similar steps to control Beri-beri. In this instance barley was sub-

stituted for polished rice, Beri-beri disappearing from Japan's navy very shortly thereafter.

With the advent of Eijkman's work, in 1897, on the disease Beriberi, the first positive step along the lines of scientific investigation of this problem was taken. He noticed that in certain localities the inhabitants lived, to a great extent, on polished rice. In these places Beri-beri was very common, while in other places where the people used the whole brown rice, the disease was rare. Eijkman then proceeded to extract whole brown rice and polished rice. He fed these extracts to fowls. Those which were fed the brown rice extract did not develop Beri-beri, whereas those fed on the polished rice extract did develop it. He also found that the whole rice extract was a cure for the disease. Eijkman's final conclusion was that Beri-beri is a nutritional disease, due to deficiencies, and not due to any pathogenic bacteria in the body. This man was a pioneer in the experimental study of these mysterious food factors.

Name Vitamin Proposed

The next great step came when Dr. Casimic Funk repeated Eijkman's work and proposed the name vitamin.

Five vitamins have been discovered, to date, namely vitamins A, B, C, D, and E. The best indication of the properties, or effects, of these food factors is embodied in the descriptive adjectives anti-conjunctivitic, anti-neuritic, anti-scorbutic, anti-rachitic, and anti-sterile, respectively.

Vitamin A, the anti-conjunctivitic vitamin, is present in butter fat, cow's milk, egg yolk, and cod liver oil in comparatively large quantities[1]. An interesting fact is that if the cow eats vitamin rich food, her milk is vitamin rich.

Lack of this food factor causes a disease known as conjunctivitis, xerophthalmia. Its manifestations are dryness of the eye ball, excessive redness, and much irritation. Lee says that this is a disease produced by pathogenic bacteria[2]. Xerophthalmia is cured by feeding the patient substances rich in vitamin A.

Heat Destroys Vitamin A

Heat destroys this nutritional factor in the presence of air. In an inert atmosphere, it will withstand high temperatures.

It has been found that the radiant energy of the sun delays the onset of xerophthalmia in rats[3].

Cooking and canning processes do not have any effect on vitamin A[4], whereas ultraviolet light destroys it[5].

This vitamin is soluble in fats. Drummond has shown that the hydrogenation of oils which contain it causes its destruction. Ozone inactivates oils which contain it.

A relationship seems to exist between pigmentation and vitamin A content[6]. For example, very yellow butter fat contains a larger quantity of this food factor than the paler butter fats. Yellow corn likewise has a high vitamin A content. In green vegetables and leaves, where vitamin A is found in high concentrations, there is a yellow pigmentation which is masked by the green color of the plant.

Passing on to vitamin B and its characteristics, we find that it is the anti-neuritic vitamin. Its lack causes the polyneuritic disorder

[1] Numbers refer to bibliography to be published next month.

called Beri-beri. Beri-beri is an acute disease, characterized by cachexia, and inflammatory changes in the nerves.

Upon feeding animals that have this disease, with foodstuffs rich in vitamin B, they recover. A normal diet sufficient in B content prevents Beri-beri, and aids growth.

Autopsies performed after death due to a deficiency of this vitamin reveal the following facts:

1. Most of the organs have ceased to develop.

2. The stomach, liver, pancreas, and the thyroid glands have wasted away and ceased to develop. This is especially true of the reproductive organs.

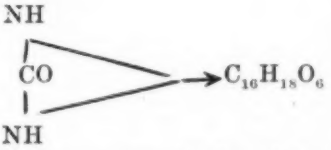
Sources of Vitamin B

Diets rich in yeast and wheat bring back all of these organs to their normal condition and they then function as well as they did originally. Yeast is the richest source of vitamin B, the wheat embryo ranking second. It is also found in most seed embryos, leafy plants, vegetables, and fruit juices.[1]

Vitamin B cannot be stored in the body so well as can the fat soluble vitamins. This vitamin is soluble in water and in alcohol.

Vitamin B is not a colloid, for it can be dialyzed. It is more easily destroyed in alkaline, than in acid or neutral solution. Chamberlain and Vedder, U. S. Army surgeons, have found that this vitamin is not a sugar, a salt, an alkaloid, an organic phosphorus compound, or an amino-acid. They concluded that the substance is an organic base, but not an alkaloid.

Funk, who claimed to have isolated this food factor, thought that it was a pyrimidine base. He went so far as to assign a formula to it, i. e.:



No evidence can be found to support this conclusion.

W. H. Eddy believed that vitamin B might be one of the bioses. In this connection it may be restated that Chamberlain and Vedder found that it was not a sugar. Eddy suggested further that the substance is crystalline, and melts at 223° centigrade.

Vitamin B can be removed from orange juice by shaking with fullers' earth. This is clearly a case of adsorption.

A deficiency of this vitamin affects the nitrogen metabolism of rats. It causes the appearance of creatinuria, accompanied by a slow wasting away of the skeletal muscles.

Uhlman suggests that vitamin B acts as a catalyst in stimulating the endocrine glands.[6]

The Anti-Scorbutic Vitamin

Vitamin C is the anti-scorbutic vitamin. Its presence in our foodstuffs prevents scurvy, and conversely, its absence causes scorbutus.

Scurvy is a skin disease characterized by livid spots, spongy gums, and bleeding from the mucous membranes.

Foodstuffs rich in this food factor are: raw cabbage, white lettuce, orange juice, lemons, citrus fruits, and tomatoes. Cereal grains in the dry state are practically devoid of vitamin C. When the seeds are caused to germinate, vitamin synthesis begins; Vitamin

C then puts in its appearance.

Vitamin C is quite readily destroyed when heated in solution, and also in the presence of oxygen. When air is bubbled through orange juice its vitamin C efficiency is destroyed. Acids protect it and alkalies destroy it. Vitamin C is not colloidal in nature, for it can be dialyzed.

This nutritional factor cannot be stored in the body to the same extent as vitamin A.

Apples canned with their normal oxygen content lose all detectable amounts of vitamin C. The same effect is noticed when the oxygen is replaced by nitrogen. But, when apples are exhausted of their oxygen content, they lose no detectable amount of their vitamin C content.[7]

Ordinary light is unnecessary for the synthesis of this vitamin. Ultraviolet light has been found to be of no aid in its synthesis.[8]

Broken femurs, in guinea pigs, showed no signs of healing in three to four weeks when there was a deficiency of vitamin C. When they were fed a normal amount of the vitamin, the bone was completely consolidated in five to six weeks.

The metabolism of the cholesterol of the suprarenal glands is profoundly disturbed by a deficiency of this nutritional factor.[10]

Vitamin D Anti-Rachitic

Vitamin D is the anti-rachitic vitamin. Like the foregoing three vitamins it is both a preventive and a remedy.

Rickets is a children's disease. It is characterized by the failure of the bones to ossify. The bones become soft and bulky. Calcium, in the form of the oxide, and phosphorus are not taken into the bones when vitamin D is absent. This causes rickets.

It has been found that the mineral matter in rachitic bones falls from the normal 63% all the way down to 25%, in many cases. These figures, of course, do not apply to all cases of rachitis. Cod liver oil, although containing very little mineral matter, completely rectifies this condition. The vitamin investigators wonder at this and ask the question, "How can cod liver oil, which contains so little mineral matter, put so much of it into the bones?" They then proceed to say, in answer to the question, that vitamin D in the oil stimulates the bones so that they utilize the mineral matter in the blood.

It has been shown that, in the absence of vitamin D, the body cannot use its own rich deposits of calcium, and the calcium piles up in the system.

Curative Effects of Light

Hess came to the conclusion that ultraviolet light possesses a curative effect in rachitis like that of vitamin D. Steenbock went further and showed, experimentally, that foods which allow rickets to develop, act in just the opposite way after treatment with ultraviolet light. He has shown that irradiation with ultraviolet rays causes inactive vegetable oils to acquire anti-rachitic potency. The anti-rachitic factor has been found to multiply eight times when foodstuffs containing it are exposed to ultraviolet light.

Further, sawdust which has been irradiated with this short-wavelength light has been found to acquire anti-rachitic properties.

N-butyl nitrite, when heated with cod liver oil, rich in vitamin

D, destroys its anti-rachitic potency[12].

Cholesterol seems to be linked closely to vitamin D. If we saponify cod liver oil with sodium hydroxide, and remove the soap that is formed, it will be found that the cholesterol, which is left behind, contains almost the entire vitamin content of the oil. Many other widely differing experiments support this view.

Cod liver oil varies in vitamin D content with the diet of the cod and the treatment of the oil.

Passing to consideration of vitamin E, we find that it is the anti-sterility vitamin. H. M. Evans first clearly established its existence. He separated it from vitamin B, and named it vitamin X. Later it became known as E.

Sources and Effects

This vitamin is found in lettuce, meat, wheat germ, oats, and alfalfa.

Sterility is induced by a deficiency of this accessory food factor. In cases where it has been so induced, the feeding of vitamin E returns the individual to normal reproductive powers.

Vitamin E is soluble in many fats and so-called fat solvents such as chloroform and benzene. It is not destroyed by heat, and it can be distilled at 233° Centigrade. Hydrogenation does not destroy this vitamin.

It has been found that the irradiation of foodstuffs, or of the individual, with ultra-violet light does not compensate for vitamin E deficiency.

The suggestion, found under the discussion of vitamin B, concerning its stimulating power for the endocrine glands, probably does not apply to that vitamin. Since that work was done, the fifth vitamin has been isolated. Since this fertility factor was intimately mixed with B, it therefore seems necessary to assume that the stimulation noted was due, not to vitamin B, but to vitamin E.

Characteristics Common to Vitamins

There are some characteristics common to all of the vitamins. All of the vitamins are growth promoting. An excess intake of them does absolutely no harm. Furthermore, they are not habit forming like certain drugs and alkaloids. Careful studies have brought vitamin investigators to the conclusion that plants are the only organisms that can manufacture them, but that animals can store them. Germinating seeds contain vitamins by virtue of synthesis, while dry seeds do not contain them. It also seems that seeds that germinate in the light are more productive of these nutritional necessities, than are those that germinate in the dark. It is believed that vitamin synthesis is related to the green coloring, or chlorophyll part of the plant. And, quantitatively, the greater the green surface, the greater the vitamin producing power of the plant.

A good definition of vitamins is given by Madame Randoin[14]. It is in accord with the lack of definiteness which enshrouds the subject. She says, "Vitamins are defined as substances, still indeterminate chemically and physically, which the animal organism cannot synthesize, and which possess properties recognized in certain fractions of the unknown part of the diet, which are indispensable for the accomplishment of vital phenomena

in the adult, or during the course of the development of the animal, and whose absence produces characteristic pathological nutritional changes."

Some of the data given in this brief survey of the vitamins is quite contradictory. This is due to the fact that the vitamin investigators are not yet sure on all matters pertaining to the subject.

(To be continued)

New Trade Marks

We have arranged with the National Trade-Mark Company, 635 F Street, N. W., Washington, D. C., to conduct this department for our readers. The trade-marks have recently been passed for publication by the United States Patent Office and are in line for early registration unless opposition is filed. For further information address National Trade-Mark Company.

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WHITE HORSE. No. 265,317. Charles H. Briggs, Chicago, Ill.; for antiseptic chemical cleaner for cleaning the hands, floors, wash rooms, toilets, sinks, ice boxes, stoves and metals.

DRI CLENS ALL. No. 265,866. Warren Soap Manufacturing Co., Boston, Mass.; for dry cleaning soap preparation.

PADRE. No. 267,505. The Palmolive Peet Co., Chicago, Ill.; for soap.

Oil Firms Consolidate

AT the recent annual stockholders' meeting of the Chickasha Cotton Oil Co. the acquisition of the Anadarko, Mangum and Hollis cotton oil companies was approved. The directorate of the Chickasha firm was increased from ten to eleven in order to elect the general manager of the Anadarko company to the board. It is expected that earnings of the Chickasha plants now will be better than $5.50 a share.

Glidden Moves Offices

The New York office of the Glidden Food Products Company, now at 82 Wall Street, will be located at 209 14th Street, Long Island City, after October 1. The Glidden Company of Cleveland purchased the new location recently as a permanent home for its New York offices and warehouses and for its Purit department, which markets the product of the Purit Company of Amsterdam, Holland.

Lauro Writes Paper

M. F. Lauro recently wrote a paper on the subject of separation and identification of various fatty acids by the determination of solubilities of their metallic soaps in various solvents. The paper was prepared by Mr. Lauro as a thesis for his M. S. degree at the Brooklyn Polytechnic Institute, New York.

This paper will be published in full or abstract in OIL & FAT INDUSTRIES in an early issue.

The Determination of Gossypol Structure

BY E. P. CLARK *

U. S. Dep't of Agriculture, Washington

THE next phase of the problem undertaken was that of obtaining information which might lead to the determination of the structure of gossypol. Work of this nature has previously been limited to the experiments of Carruth (3) who studied the effects of alkali fusion and several oxidizing agents upon gossypol but did not succeed in obtaining any pure crystalline derivatives or other evidence of a clarifying character.

Among many possibilities which presented themselves was the action of hot concentrated alkali upon gossypol (13). This reagent, forty per cent sodium hydroxide, at the temperature of the steam bath, brought about a well defined transformation. When the two components are first brought together, the gossypol does not immediately dissolve as it does in less concentrated alkali, but forms a dark orange paste. If this is continuously heated at the temperature of the steam bath and frequently stirred, a reaction slowly takes place in which the color of the mixture becomes gray.

The products of the reaction are formic acid and a new phenolic substance which has been designated as apogossypol. Apogossypol is a colorless crystalline material having no definite melting point. It is soluble in ordinary organic solvents, and the solutions thus formed darken more or less quickly depending upon the nature of the solvent employed. It dissolves freely in dilulte alkali, from which it is precipitated by carbon dioxide. Its alkaline solutions darken at once and rapidly acquire a purple color. Even crystalline apogossypol is so unstable that a few hours' exposure of the dry crystals to air and light causes them to change to a jet black powder. Another characteristic of its phenolic nature is the bluish green color reaction produced when a dilute alcoholic solution of the substance is treated with ferric chloride.

Apogossypol Derivatives

Apogossypol readily forms colorless crystalline acetyl and methoxy derivatives, which are, in contrast with the free substance, quite stable. A study of these substitution products, as well as of the free phenol, has shown that apogossypol has the molecular formula $C_{28}H_{30}O_6$. Furthermore, it has been shown that there are six acetyl groups in the acyl derivative. No direct determination of the methoxy groups in the methyl ether could be made since the substance was completely inert in boiling hydriodic acid under the conditions used in the Zeisel method. Carbon and hydrogen figures, however, checked well for a hexamethyl ether. It therefore follows that all the oxygen atoms in apogossypol are present as hydroxyl groups. From a consideration of the molecular formula for gossypol, $C_{30}H_{30}O_8$, it will be seen that strong hot alkali, under the conditions of the

*Continued from the August issue of OIL AND FAT INDUSTRIES.

experiment, functions only in removing the two carbonyl groups from the gossypol molecule. Since formic acid is the only other substance produced by the reaction, the carbonyl groups must have been eliminated as formic acid. Such a process requires that each mol of gossypol should produce one mol of apogossypol and two mols of formic acid. The yield of each of these substances actually obtained corresponded to 97.5 per cent and 95.3 per cent respectively of that demanded by the theory.

Nature of Chromophores

It is noteworthy that in the elimination of the two carbonyl groups from gossypol the characteristic yellow color of the substance disappears. This strengthens the hypothesis previously advanced that the chromophores of gossypol are carbonyl groups.

The toxicity of apogossypol was found to be approximately one third of that of gossypol. The lethal dose, when given intraperitoneally in oil solution to white rats, is from sixty to seventy-five mg. per kilogram of body weight. Its physiological action, however, is in sharp contrast with that of gossypol as no chronic effects follow the administration of small doses. Its action seems to be restricted to the development of acute toxic symptoms only.

Experiments upon the oxidation of gossypol (14) as a whole have been more or less discouraging, for with most reagents and methods tried either nothing or only amorphous or tarry products, having no tendency to crystallize, were obtained. With potassium permanganate, however, certain products were obtained that have a significant bearing upon at least one phase of the problem. In these experiments gossypol dissolved in slightly more than 2 mols of sodium hydroxide was treated at 0° with potassium permanganate in the proportions of one part of gossypol to five parts of permanganate. The reaction was decidedly energetic and terminated in a short time. The manganese dioxide formed was removed by filtration, and the filtrate was made acid with sulfuric acid. In adition to the evolution of carbon dioxide there was evidence of the presence of volatile fatty acids in the reaction mixture. The solution was therefore steam-distilled, and the distillate was examined by the method of Dyer (15) for fatty acids. From an examination of the distillation curve obtained it was possible to show that formic, acetic and isobutyric were the acids present in the mixture. These conclusions were verified by applying specific confirmatory tests and by preparing certain characteristic derivatives of the respective acids. It was further demonstrated that the acids mentioned were the only ones present. A quantitative estimation of the different acids was made, and the yield obtained in the case of isobutyric acid was 92.5 per cent of that required by the theory if it is assumed that each mol of gossypol yields one mol of isobutyric acid.

Evidence Supporting Side-Chain Theory

The existence of formic acid under the conditions of the experiment may appear at first to be paradoxical; but when it is considered that the potassium permanganate was not used in excess, it is evident that some of this acid could easily escape oxidation. It would be unwise at this

time to attribute the formation of formic and acetic acids to any definite structure in the original material, but the identification of isobutyric acid as a decomposition product seems to indicate that there is a side chain in the gossypol molecule involving at least the group $(CH_3)_2{=}CH{-}\overset{1}{C}{-}$.

Oxidation Products

From the results obtained in attempting to oxidize gossypol it was soon realized that in order to progress in the degradation of the molecule, the hydroxyl groups would have to be protected. There are available at the present time three derivatives fulfilling this condition, namely, hexaacetyl gossypol, hexaacetyl apogossypol and apogossypol hexamethyl ether. These materials were subjected to oxidizing agents under various conditions and to date three different products have been obtained in analytically pure crystalline form. The oxidizing agent used in the preparation of each was chromic acid, but the conditions imposed in the various cases were different.

The product obtained from hexaacetyl gossypol is a bright yellow neutral compound which possesses no carboxyl or free hydroxyl groups and which has the molecular formula $C_{33}H_{30}O_{12}$. It is a tetraacetyl derivative of a substance $C_{25}H_{22}O_8$. It follows that in its formation two acetyl groups have been replaced by two quinone groups and that the substance has a smaller molecular weight by C_2H_8 than gossypol. The carbonyl groups originally in gossypol apparently have been retained in the new substance, as aniline condenses with it in much the same manner as with gossypol. This is not the case with the quinones to be immediately described which are obtained from apogossypol.

A product obtained by the chromic acid oxidation of hexaacetyl apogossypol is a bright yellow neutral substance having a melting point of 230° and the molecular formula $C_{30}H_{28}O_{10}$. It is a tetraacetyl derivative of a compound $C_{22}H_{20}O_6$. In this case two acetyl groups have also been replaced by two quinone groups, and the original apogossypol has lost C_6H_{10}.

The oxidation of apogossypol hexamethyl ether gave a bright yellow neutral crystalline compound $C_{32}H_{24}O_8$. It was impossible to determine the methoxy groups in this preparation, as boiling hydriodic acid under the conditions used in the Zeisel method had no appreciable action upon the substance; but from analogy it appears that two methoxy groups have been replaced by quinone groups and at the same time two hydrogen atoms have been oxidized to quinone groups. If this is the correct explanation of the mechanism of the reaction, the new substance would be a tetramethyl ether of a tetra quinone $C_{28}H_{26}O_8$.

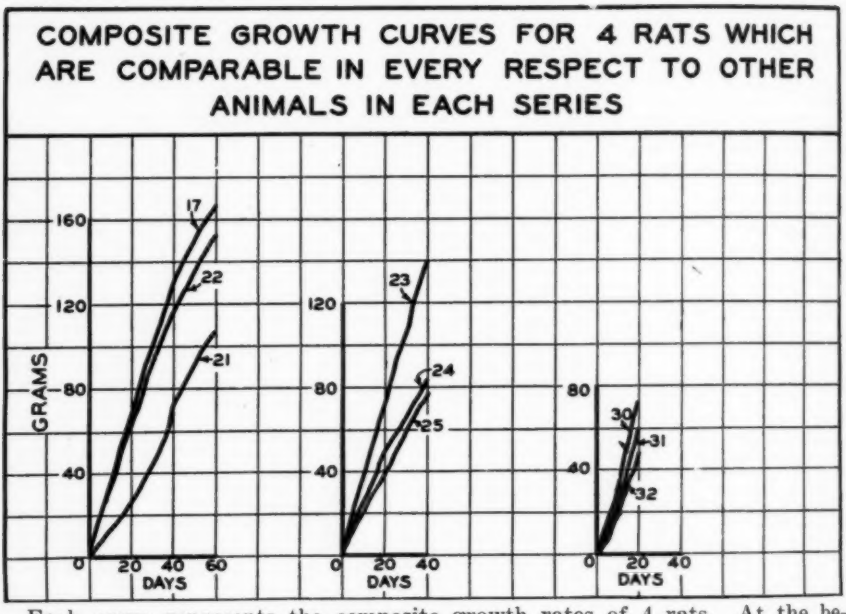
Biological Experiments

The foregoing information summarizes the definite chemical facts thus far obtained. The nature of the results precludes the possibility of making a statement regarding the nature of the gossypol molecule; but the experience gained by working with the material has indicated a method by which the problem of cottonseed injury may be attacked. Work in this direction is now in progress, but as it is necessary in such investigations to resort to animal experimentation, attention was directed to the choice

of suitable animals for the purpose. Since the Protein and Nutrition Division of the Bureau of Chemistry and Soils, United States Department of Agriculture, has been engaged in and is equipped to carry on such studies, their cooperation, in the personnel of Dr. E. M. Nelson and Dr. D. B. Jones, was secured.

In this work, as in the study of nutritional problems in general, it was desirable to employ small laboratory animals for the necessary tests. The use of such animals would offer the advantage of economy in labor and expense; and also because of the character of the material to be investigated, their use would insure more dependable results. The albino rat is the animal best suited for this purpose, but certain workers have conveyed contradictory impressions regarding its susceptibility toward orally administered gossypol.

Our first problem was therefore to establish to what extent results obtained on rats could be applied to gossypol poisoning studies. Some experiments along this line will be briefly described. Five groups of rats received a basal diet containing graduated quantities of pure gossypol; a sixth group, used



Each curve represents the composite growth rates of 4 rats. At the beginning of the experiment all the animals weighed approximately 40 gm.

17—Controls on basic ration.

21—Basic ration plus 0.1 per cent gossypol.

22—Basic ration plus 0.05 per cent gossypol.

23—Controls.

24—40 per cent raw cottonseed flour, gossypol free.

25—40 per cent cottonseed meal flour, 1.6 per cent bound gossypol.

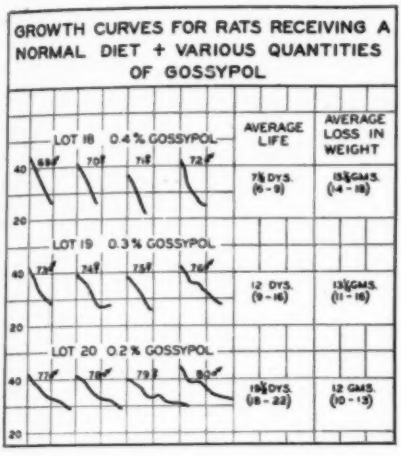
30—Controls.

31—40 per cent raw cottonseed flour, gossypol free, with modified salt mixture.

32—40 per cent cottonseed meal flour

as controls, received the same diet without gossypol. The animals in all the groups were comparable as to weight, sex, and parentage, and the basic diet was adequate in every known factor necessary for an optimum rate of growth.

The growth curves were surprisingly uniform and showed that even 0.05 per cent of pure gossypol produced a noticeable retardation of the growth rate. One tenth of one per cent gossypol produced much greater depressions, and 0.2



per cent gossypol, or more, in the ration caused loss in weight and death. The time until death occurred, when lethal quantities of the substance were consumed, had a direct relationship to the dose. These findings are recorded graphically in the accompanying charts.

Animals receiving a ration in which forty per cent of the normal diet was replaced by cottonseed flour to serve as the sole source of protein grew more slowly than the controls. In one experiment raw cottonseed flour freed from gossypol was used, and in another experiment cottonseed meal flour containing no free gossypol, but having 1.6 per cent bound gossypol was fed. The animals on the raw cottonseed diet grew a little more rapidly than those on the cottonseed meal preparation. The difference between the growth rates of animals receiving cottonseed flour and the controls was in part due to an improper mineral balance. When this factor was corrected in another set of experiments the growth rates of the animals on the modified diets were better than those of the previous set but they were still below those of the controls. Here again the animals receiving cottonseed meal made the least progress. These results are shown by the accompanying curves.

Although the technique of testing the toxicity of cottonseed meal products has not yet reached the desired degree of perfection, it is hoped that practical information resulting from the chemical data now available upon gossypol will soon be forthcoming.

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The American Oil Chemists' Society

Notes and Correspondence

A. O. C. S. Fall Meeting

THE Fall meeting of the American Oil Chemists' Society will be held in the Board of Managers' rooms of the New York Produce Exchange on October 25 and 26. W. L. Beatty, President of the Produce Exchange, will welcome the Society. The Governing Board of the Society determined to hold this meeting after considering the excellent attendance of the first one held in New York last year, and selected New York for the gathering place due to the city's central location for oil chemists. The Board fully expects this year's fall meeting to be as successful as the previous one, and asks each member of the Society to reserve these days in order to assure himself the opportunity to benefit from the combined ideas and experience of the majority of the chemists connected with the Society.

Standardization of Lovibond Glasses

(Investigation by the Bureau of Standards in Cooperation with the American Oil Chemists' Society.

FINAL REPORT

THE cooperative arrangement whereby the A.O.C.S. supported a Research Associate at the Bureau was terminated July 2nd, when Miss Walker was transferred from status of Research Associate to that of a regular Bureau employee. However, calibration of red glasses already on hand with Mr. Putland was continued and completed during July. Final report on the value of these glasses was made to Mr. Putland and all remaining glasses were returned July 31st. The following table gives a summary of glasses calibrated under the co-operative arrangement:

Red Glasses	
Reported in OIL AND FAT INDUSTRIES, Jan., 1928.....	129
Reported to Mr. Putland.....	409
Yellow Glasses	
Final report now about ready (Abstract included in report for June)	65
Total	603

Paper by Judd on "Effect of Temperature Change on the Color of Red and Yellow Lovibond Glasses" is now in course of publication in the Bureau of Standards' Journal of Research.

Report on "Calibration of Sixty-five 35-Yellow Lovibond Glasses" is being prepared for publication in the Bureau of Standards' Journal of Research.

Considerable progress has been made on reducing the data taken at New Orleans in May on tests of color sense and sensibility to differences in Lovibond red.

IRWIN G. PRIEST

Correction

In the August issue of OIL AND FAT INDUSTRIES, the statement was made, in the article entitled "1928-29 A. O. C. S. Committees Announced," to the effect that the Committee on Analysis of Commercial Oils and Fats and the Basic Research Committee had been abolished because of completion of work. These committees, however, are still operating, but under the auspices of the American Chemical Society and the Interstate Cottonseed Crushers' Association, respectively.

New Forbes Laboratory

L. B. Forbes Laboratories, Inc., has opened a new laboratory at Little Rock, Ark., under the direction of Edgar H. Tenent. Mr. Tenent resigned his position with the International Sugar Feed Company at Memphis, with whom he has been connected for the past five years, in order to assume the management of the new Forbes laboratory.

Cotton Distribution

A preliminary report by the Department of Commerce on the distribution of cotton during the year ending July 31, 1928, shows that 16,793,300 bales were available during the year. Exports amounted to 7,530,879 bales, domestic consumption to 6,832,689 bales, and stocks on hand to 2,531,582 bales. During the year 20,000 bales of ginned cotton were destroyed. Aggregate distribution amounted to 16,915,150 bales, or 121,850 bales in excess of supply.

Referee Applicants

Mr. H. M. Shilstone has made application for Referee Chemist certification, according to word received from Mr. J. C. P. Helm, secretary-treasurer of the American Oil Chemists' Society. (First publication.)

Abstracts

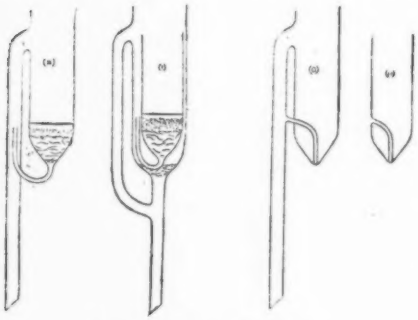
DETECTION OF HARDENED FAT IN BEEF FAT FROM THE IODINE VALUE OF THE SOLID FATTY ACIDS SEPARATED BY TWITCHELL'S METHOD. S. C. L. GERRITZEN and M. KAUFFMAN. (*Chem. Weekblad*, 24, 554-556.)—Twitchell's method (*Analyst*, 46, 466) has been found suitable for the separation of solid fatty acids from hardened oils. The iodine values of the acids thus obtained from beef fat are usually less than 5, and a value greater than 6 indicates the presence of a foreign substance. For mutton fat the values vary from 1.7 to 12.5 and this probably indicates the incomplete separation of the fatty acids, or the presence of solids, unsaturated fatty acids. The percentage of isooleic acid may be calculated from the expression $-95.S.(I-5)/9{,}000$, where S is the amount of sample taken, and I the iodine value of the solid, saturated fatty acids. For butter-fat, lard and hardened whale oil the values vary from 4.6 to 5.3, 1.1 to 3.1, and 6.2 to 27.3, respectively. By the addition of known amounts of hardened whale oil to beef fat, the proportion present in a given sample may be determined from a comparison of the iodine values of the separated fatty acids (*cf. Analyst*, 49, 460).

PHYTOSTERYL ACETATE TEST AND THE PHYTOSTEROL OF SUNFLOWER SEED OIL. J. ALLAN and C. W. MOORE. (*J. Soc. Chem. Ind.*, 46, 433-434T.)—The value of the phytosteryl acetate test, when applied to the detection of animal fats in fats of vegetable origin, has been diminished by the discovery of phytosterols which form acetyl derivatives having melting points ranging from 115° to 250° C., although values below 125° C. are infrequent. It is, however, now found that a phytosterol, $C_{29}H_{49}OH$, occurring in normal amount in sunflower seed oil and constituting the bulk of the phytosterols present, gives an acetyl compound melting at 119-119.5° C. (corr.). Mixtures of this acetyl derivative with those of certain other phytosterols melt at temperatures ranging from 121° to 123.5° C. (corr.), whilst a mixture with an approximately equal weight of cholesteryl acetate shows abnormal behavior and melts at 121-122° C. (corr.). The difficulty which thus arises in using the phytosteryl acetate test for examining sunflower seed oil or its mixtures for animal fats, is also encountered with hydrogenated sunflower seed oil. The sterols obtained from several other important edible vegetable oils examined yield acetyl derivatives with melting points lying between 122° and 131° C.

Continuous Extractor for Rapid Work

DOUGLAS HENVILLE, in *The Analyst* for July, 1928, reports the development of a continuous extractor. "This apparatus," he writes, "was devised for the rapid extraction of large quantities (50 grms.) of materials from which the oil or other soluble substance can be easily extracted, such as the extraction of phenols from carbolic powders having a siliceous base.

"The bottom of the extractor is packed with cotton wool or other suitable filtering medium. It is preferable to use one complete piece of cotton-wool, as, if several layers be used, air is enclosed between them which may rise during the extraction and disturb the packing. A weighed quantity of material is introduced, and the extractor attached to a reflux condenser. A flask containing the solvent is fitted to the stem of the extractor. The flask must not contain so much solvent that the extractor could fill up and overflow through the large side tube into the flask. The solvent is distilled into the extractor in the usual way.



Types of new extractor

"Where the extract is heavy, as in the case of the extraction of phenols, etc., or where a considerable depth of a fine powder is being extracted, a certain amount of 'head' is at first necessary to drive the extract over and distillation is continued until a rapid flow of solvent is obtained; in addition, air bubbles have a retarding effect, but these gradually disperse.

Space Left to Develop Head

"A large margin of space to allow for the formation of this 'head' has been allowed in the design of the apparatus. The 5 grm. size extractor has also been made of such a diameter that the 3 to 5 grm. quantities usually taken for extraction form a shallow layer and do not obstruct the passage of the solvent.

"The drawing (X) shows the complete extractor and the drawing (O) shows another type in which the small tube is placed inside.

"The apparatus has the following advantages:

"1. Neither thimbles nor filter paper packets are necessary (they can be used if required).

"2. The substance to be extracted is always totally immersed in the solvent.

"3. The 'pressure' is directly on the solvent in contact with the material and the extracting liquid is always in moton. (In the Soxhlet extractor the 'pull' acts on the liquid outside the thimble. After each 'siphoning over' a little more extract-containing solvent drains from the thimble into the bottom, and the extractor has to be filled again before 'siphoning' takes

place. This continues until extraction is complete.)

"4. There is a continuous and rapid flow of solvent. The rate of flow is, of course, dependent on the rate of distillation.

"5. If the apparatus be left to 'stand overnight,' the substance remains totally immersed, and when the extractor is started again the first few ml. of solvent from the condenser cause circulation to continue.

"6. As soon as the solvent is circulating easily, only a few ml. are necessary in the flask to continue the extraction.

"7. The contents of the extractor are visible.

"The extractor may be used for the extraction of substances which require no filtering medium, and cakes, etc., in which some form of filter is necessary.

"Using asbestos as a filtering medium, cocoa has been successfully extracted without any of the fine powder passing through into the flask.

"On the same principle (1) and (2) are glass thimbles which are placed in the container shown. The thimble may rest on cotton-wool, which supplies a further filtering medium, or may rest directly on the glass, when the hot vapor will pass up and around the thimble.

"Except for special purposes, when these thimbles might be required, the complete extractor (X), as shown in the accompanying diagram is simpler and more satisfactory in use."

Book Reviews

THE INDUSTRIAL CHEMISTRY OF THE FATS AND WAXES. By T. P. HILDITCH, D. Sc., F. I. C., Professor of Industrial Chemistry (Oils, Fats, and Waxes) in the University of Liverpool, with an introduction by E. Frankland Armstrong D. Sc., LL. D., F. R. S., 8vo., XV—461 pp. D. Van Nostrand & Co., New York.

This admirable work offers what appears to be the most logical concise discussion of the chemistry of oils, fats and waxes from the viewpoint of advanced chemical knowledge, which has come to our notice.

The word "oil" is omitted from the title with purpose, as the author suggests the logical substitution of the term "liquid fat" for "fatty oil" to avoid confusion with oils of petrolic origin. An excellent suggestion, but one which is almost impossible of practical attainment, since its general adoption would involve a radical change in the speech-habits of all persons connected with a vast, world-wide industry. For purely chemical use, however, it would promote clarity.

The author divides his work into ten sections, progressing from the chemical nature of fats and the composition of natural fats through the technology of fat production and utilization. The outstanding chapters are those on the composition and properties of fatty acids, on the distribution of fats and waxes in nature and on the physical chemistry of soap. As would be expected in a work from the pen of so eminent an educator (and in consonance with the purpose of the book, as expressed in the preface), the purely chemical chapters, including those on analysis, as well as those on composition and theory, surpass the technologic sections in

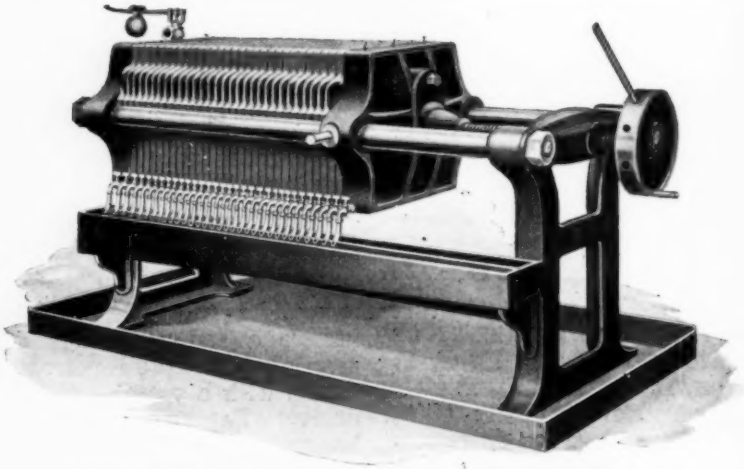
detail of material, but the latter are characterized by a summation of preferred practices and by a lack of misinformation which are most refreshing when contrasted with some previous efforts to cover the same ground in English.

The classification of the bibliography in sections is of extreme value.

Summarizing, here is a work on the chemistry of fats and waxes which is modern in every sense of the word, and which is free from glaring errors of fact or of typography.

It will provide an ideal textbook for advanced students, and it is worthy of a prominent place in the reference library of every chemist or chemical engineer who is engaged with or interested in fats, waxes, or soaps.

A. P. L.



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